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- (71) Applicant Procter & Gamble Company (USA-Ohio), 301 East Sixth Street, Cincinnati, Ohio 45201, United States of America
- (72) Inventors Stephen Henry Iding Robert Henry Callicott
- (74) Agent and/or Address for Service Maxim Courtney Brooks, Procter & Gamble Limited, Whitley Road, Longbenton, Newcastle upon Tyne NE12 9TS

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(54) Synthetic surfactant flakes

(57) Hot surfactant flakes are made from drum drying a water-wet paste containing sodium alkyl sulfate (AS), sodium alkyl benzene sulfonate (LAS), and sodium chloride. The hot flakes are cooled in a low moisture environment having a dewpoint of up to 4°C, e.g., under a dry nitrogen or dry air blanket. The low moisture environment prevents hydration and stabilizes the flakes. The flakes can be used to make surfactant cakes. Cakes made with the flakes of this invention can contain large amounts of perfume.

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Improved synthetic surfactant flakes and process for making them

5 5 TECHNICAL FIELD The present invention relates to a process for making drum dried synthetic surfactant flakes. Synthetic surfactant flakes are a key ingredient to making surfactant cakes for automatic toilet bowl cleaning products. Such cakes are particularly useful in conjunction with a toilet tank dosing dispenser which automatically dispenses a ration of surfactant, perfume, and/or dye, and 10 10 optionally other ingredients to the bowl of a toilet, responsive to the flushing of the toilet. BACKGROUND The technology of drum drying wet synthetic surfactant materials is old. Sodium alkyl benzene sulfonate (LAS) is a notoriously hygroscopic material. Substantially pure LAS flakes are tacky. 15 Sodium alkyl sulfate (AS) flakes are free flowing and have noncaking properties. Mixtures or co-15 flakes of AS/LAS have varying physical properties. U.S. Pat. No. 4,253,993, J. C. Ramsey and P. J. Schoner, issued March 3, 1981, for Shampoo in Flake Form, discloses a process comprising drum drying an aqueous slurry of 45-75% sodium alkyl sulfate (AS), monoethanol amide (MEA), sodium sulfate to make a flake 20 containing 40-60% AS, 2-5% MEA and 20-50% sodium sulfate. Although other drying 20 techniques are disclosed, this patent does not teach the use of nitrogen or dry air to cool the drum dried flakes. U.S. Pat. No. 3,950,275, Toyoda et al., issued April 13, 1976, discloses the use of a coating of builders to stabilize spray dried granules of hygroscopic LAS detergent compositions. This patent is cited to show the state of the art. In the food art low humidity cooling of drum dried food flakes is known. The following 25 25 references are examples. "Improved drum-dried tomato flakes are produced by a modified drum dryer" which employs low humidity collection zones. M. E. Lazar and J. C. Miers, August, 1971, Food Technology, Vol. 25, p. 830. "Secondary drying of drum-dried thermoplastic foods," M. A. Lazar and T. Rumsey, 1976, J. of Food Sci., Vol. 41, p. 696, is another 30, reference, United Kingdom Pat. Appln. 2,083,188, J. F. Fuller, March 17, 1982, discloses that a puree of fresh fruit is dried on a drum to produce flakes, the whole process being carried out under dehumidified atmospheric conditions. It is believed that the prior art does not teach stabilizing drum dried hygroscopic AS/LAS surfactant flake compositions with dry air or nitrogen. Nor does the prior art teach that such 35 AS/LAS coflakes can carry more perfume in solid cake compositions than cakes made with 35 separate AS and LAS flakes, as well as AS/LAS coflakes cooled in an environment having a dewpoint over 4°C. This invention relates to surfactant flakes which can be used to make surfactant cake compositions which are used in automatic dispensing devices. Examples of such cakes are 40 40 disclosed in U.S. Pat. No. 4,308,625, Kitko, issued January 5, 1982; U.S. Pat. No. 4,310,434, Choy and Greene, issued January 12, 1982; and U.S. Pat. No. 4,278,5671, Choy, issued July 14, 1982, entitled "Surfactant Cake Compositions;" all of which are incorporated herein by reference. The surfactants provide cleaning and sudsing in the toilet bowl and also serve to dispense other components of the composition such as dyes, perfumes, 45 organic resins, etc. Anionic surfactants, especially the organic sulfates and sulfonate types, are 45 used in these compositions because of their availability, low cost and excellent cleaning and dispensing properties. Water-soluble inert salts such as alkali metal chlorides and sulfates are used in such compositions to act as a "filler" so that the composition can be formed into cakes of desirable 50 size without using excessive amounts of active ingredients. The predominant ingredients of the 50 cake compositions are usually the surfactant, perfume and the filler salt. Anionic, nonionic, zwitterionic or cationic surfactants are used. The surfactant or surfactant mixture should be solid at temperatures up to about 100°F (40°C). Anionics and nonionics and mixtures thereof are useful. Anionics are the most preferred. The prior art anionic surfactant cakes can be described as essentially the water-soluble alkali 55 metal salts, of organic sulfuric reaction products having in their molecular structure an alkyl or an alkylaryl radical containing from 8 to 22 carbon atoms. A major problem in this art has been short and/or erratic longevity of surfactant cakes. Another problem is related to the incorporation of higher levels of perfume into surfactant cake 60 60 formulations while maintaining desired firmness.

SUMMARY OF THE INVENTION

Hot, drum dried sodium alkyl sulfate/sodium alkyl benzene sufonate (AS/LAS) flakes are cooled in a dry gas environment at a dewpoint of 4°C or below to prevent insidious hydration and to provide improved flakes. The flakes, which are 90% to 99.5% AS/LAS surfactant, are

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with a doctor blade. The hot, dried flakes are carefully cooled in a low moisture environment, e.g., under a dry air blanket or a dry nitrogen blanket, to avoid undesirable, insidious hydration.

65 The dry air or nitrogen should have a dewpoint of 4°C or below. Such dry air can be obtained BEST AVAILABLE COPY

65 incorporated herein by reference.

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Total

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Perfume Formulas

1-B

5	Component	Weight %
•	Isobornyl Acetate	10.0
	Lavandin	15.0
	d'Limonene	20.0
10	Lemon Oil C.P.	20.0
	4-Tertiary Butyl Alpha Methyl	
	Hydrocinnamic Aldehyde	10.0
•	Methyl Heptine Carbonate	0.1
٠.	Para Cresyl Methyl Ether	1.0
15	Anisic Aldehyde	5.0
	Peppermint Oil	0.5
	Phenyl Acetaldehyde Dimethyl Acetal	2.0
	Lauric Aldehyde	1.0
	Iso Hexenyl Cyclohexenyl Carboxaldehyde	2.0
20	Methyl Iso Butenyl Tetrahydro Pyran	0.5
	Vetigreen 1% in D.E.P.	0.1
	Ethyl Methyl Phenyl Glycidate	0.8
	Diphenyl Oxide	1.0
	Musk Xylol	5.0
25	Methyl Salicylate	1.0
	1-8-Cineole	1.0
	Aurantiol	3.0
	Ligustral ·	1.0
30	Total	100.0%

Cake Firmness

The firmness of the cake is measured by the use of a penetrometer. Acceptable penetrometer readings are 100, and preferably between 40 and 80, using a Lab-Line Universal Penetrometer equipped with wax penetration needle ASTM D1321, Cat. No. 4101.

Operation:

Level base and place 100 gm and 50 gm weights on plunger top. Place bar on cut end 40 beneath penetrometer needle, raised to the zero position. Lower needle (via elevator screw) until needle just touches plug end. Depress trigger for 10 seconds (needle will lower into cake, then release. To read hardness, lower depth gauge bar until it just touches plunger.

Hardness readings are taken directly from the gauge, in units of tenths of millimeters. Penetration decreases as hardness increases.

Raise the needle to zero position, remove plug, and record plug hardness temperature.

The Salt

Sodium chloride may be included in the paste at levels of from 0.5% to 10% by weight of the AS/LAS surfactant. Its primary use is to adjust the viscosity of the paste. In the cake made from the coflakes, NaCl (salt) can be included up to about 32%, preferably 25% to 30%. About 28% total salt is optimum for the preferred cake composition which is set out in Example 11, which composition is used to evaluate the coflakes via the cake's firmness made into them. The term "salt" as used herein means NaCl unless specified otherwise.

55 The Dyes

Dyes may be included at levels of from about 0.5% to 12%, preferably 1.5% to 5%. It is highly desirable that the cakes have a pH of less than about 8.5 for dye stability. Examples of suitable dyes are Alizarine Light Blue B (C.I. 63010), Carta Blue VP (C.I. 24401), Acid Green 2G (C.I. 42085), Astrogen Green D (C.I. 42040), Supranol Cyanine 7B (C.I. 42675), Maxilon

60 Blue 3RL (C.I. Basic Blue 80), Drimarine Blue Z-RL (C.I. Reactive Blue 18), Alizarine Light Blue H-RL (C.I. Acid Blue 182), FD & C Blue No. 1 and FD & C Green No. 3. (See the patents of Kitko, U.S. Pat. No. 4,200,606, issued April 29, 1980, and U.S. Pat. No. 4,248,827, issued February 3, 1981, both incorporated herein by reference.) C.I. refers to Color Index.

65 Dispensing Means

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Dispensing means which can be used to dispense compositions of the present invention into the toilet flush water are exemplified by those described in U.S. Pat. Nos. 3,831,205, 3,341,074, 3,504,384, 2,688,754, 4,036,407, 4,171,546, 4,208,747, and 4,186,856, above noted. Details of the preferred dispensing means are disclosed in commonly owned U.S. Pat. Appln. Ser. No. 452,543, Dirksing et al. entitled "Article and Method for Maintaining 5 More Even Concentrations of Bleach in a Passive Dosing Dispenser," filed December 23, 1982, incorporated herein by reference. **EXAMPLES** 10 Preferred embodiments of the invention will be illustrated by the following nonlimiting 10 examples. In the examples below, unless otherwise stated, all AS and LAS references mean sodium lauryl sulfate, and sodium lauryl benzene sulfonate. 15 EXAMPLE 1 15 This example sets out the procedure for making stable AS/LAS coflakes. The following formula (102 kg batch) was put into a steam-jacketed crutcher with agitation and recirculation: 20 Ingredients **Parts** 20 AS (30% active)* 74.11 LAS (90% active)** 22.23 NaCl 3.18 25 NaCO₃ (25% active) 0.48 25 100.00 * EQUEX-S, manufactured by The Procter & Gamble Company, is a 29% solution of sodium 30 lauryl sulfate and 1% additional solids. Calsoft F-90 is a 90% sodium alkyl benzene powder with an average alkyl chain length of 11.3, manufactured by Pilot Chemical Co. 35 This crutcher paste consisted of: 35 Ingredients **Parts** 40 AS 22.23 40 LAS 20.02 NaCl 3.18 NaCO₃ 0.12 H₂O 51.49 45 Misc. solids from AS and LAS 2.96 45

This paste was heated to 62°C, and had a pH of about 8.7 and a viscosity which varied from 1000 to 5000.

100.00

After about 30 minutes of mixing, the paste was concentrated to about 30% moisture in a plate and frame heat exchanger and then pumped to a drum roll dryer, having a temperature of about 160°C, and dried into flakes. The flakes were cooled in a conveyor shoot under a blanket

55 of dry air having a dewpoint of less than 4°C, which was provided by a Van Air Regenerative Air 55 Dryer. The flakes had the following composition:

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	Ingredients	Parts		
5	AS	45.30		
	LAS	40.90		
	NaCl NaCO ₃	6.53		
	H ₂ O	0.23 1.00		
10	Misc. solids	6.04		1
	·	100.0		
15	EXAMPLE II The flakes of conditions using	f Example I	were agglomerated with perfume, additional NaCl, and dye at ambient wing formula:	1
20	Ingredients	Parts		2
•	Flakes	65.6		
	Perfume	11.0		
25	NaCl (added) ` Dye	21.7 1.7		2
		100.0		2
35	cm × 4.9 cm o 26°C. The strip cakes had a pH	tion was mi rifice into s is were ther I of about 9	ixed well for 7 minutes and plodded and then extruded through a 1.3 trips. During extrusion the composition had a temperature of about a cut into cakes and allowed to cool to ambient temperature. The 1.5. ge finished hardness penetrometer value of 91.	3!
40	EXAMPLE III The procedure set out in Example I is followed, except that the paste is heated to 60°C, the pH adjusted to about 8.5, the wet paste concentrated to about 35% solids, the dry roll dryer temperature is about 160°C and the hot flakes cooled under ambient conditions, dewpoint less than 4°C. The paste dried to a moisture content of about 0.8%. Solid cakes are made as in Example II. Their average cake firmness is about 80 penetrometer			
45	units.		o periodo motos.	
-	EXAMPLE IV			45
50	The same procedure set out in Example III is followed, except that the dewpoint is about 4°C. It is noted that the moisture level in the flakes of this example is less than 1.2%, even less than 1%. Yet, the flakes are inferior. Cakes made from the flakes of this example, following the procedure of Example II demonstrate an average cake firmness of above 100 penetrometer units.			
	dewpoint condit	tions for co	oples III and IV, flakes made under identical conditions, except for the oling the flakes, resulted in cakes having suitable hardness (Example eptably soft (Example IV).	
5	CLAIMS	•• •		58
0	A. forming a (a) from 20% to (b) from 20%		g surfactant flakes from a water-wet paste which is dried on a heated by:	
	(b) flave a weigr (c) from 0.5%	characterize 25°C to 9 30% of ar to 30% of nt ratio of 1	5°C water-wet paste containing: a lkali metal $\text{C}_{\text{s}}\text{-C}_{1\text{s}}$ alkyl sulfate; an alkali metal $\text{C}_{\text{g}}\text{-C}_{1\text{s}}$ alkyl benzene sulfonate, wherein said (a) and :1.5 to 1.5:1; and chloride; and	60
5	(c) flave a weigr (c) from 0.5% (d) water;	characterize 25°C to 9 30% of ar to 30% of 1t ratio of 1 to 10% so	n alkali metal C_9-C_{15} alkyl sulfate; an alkali metal C_9-C_{15} alkyl benzene sulfonate, wherein said (a) and :1.5 to 1.5:1;	60

a thickness of from 0.1mm to 1.3mm and a moisture content of up to 1.2%; and C. cooling the flakes to about ambient temperature in a low moisture environment having a dewpoint of less than 4°C. 2. A process according to Claim 1 wherein the paste contains a sodium carbonate buffer to 5 maintain a paste pH of from 7 to 9.5 in a 1% solids solution at ambient temperature. 5 3. A process according to Claim 1 or 2 wherein the roll drum dryer has a temperature of from 140°C to 190°C and wherein the dewpoint is less than 0°C. 4. A process according to any of Claims 1 to 3 wherein the heat dried flakes are cooled under a blanket of dry nitrogen or dry air. 5. A process according to any of Claims 1 to 4 wherein the alkyl sulfate and alkyl benzene 10 sulfonate have a weight ratio of from 0.8:1 to 1:0.8, preferably about 1:1. 6. A process according to any of Claims 1 to 5 wherein the wet paste is heated to a temperature of from 38°C to 66°C and concentrated to a moisture level of 30% to 40% prior to 7. A process according to Claim 1 wherein the heated roll drum dryer has a temperature of 15 from 150°C to 175°C and the flakes have a moisture content of from 0.5 to 0.8. 8. A process according to any of Claims 1 to 7 wherein the flakes are mixed with from 10% to 13% perfume, 0.1% to 5% dye, and from 0% to 30% NaCl, plodded, extruded, and formed into cakes having a hardness penetrometer value of from 40 to 80. 9. A process according to Claim 8 wherein the cake has 10% to 12% perfume and a total 20 NaCl content of from 15% to 30%. 10. A surfactant flake comprising on a weight percentage basis from 38% to 52% sodium C₉-C₁₅ alkyl sulfate (AS), from 33% to 47% sodium C₉-C₁₅ alkyl benzene sulfonate (LAS), from 0.5% to 10%, preferably from 6% to 8% sodium chloride (salt) and less than 1.2%, preferably 25 from 0.5% to 0.8% moisture, the flake having a thickness of from 0.1mm to 1.3mm, 25 preferably from 0.2mm to 0.6mm and being prepared by the process of any of Claims 1 to 9.

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